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CHARACTERIZATION OF C-55A PROPELLANT

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by

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ABSTRACT. The C-55A propellant is a cast composite utilizing a carboxyl-terminated-polybutadiene binder cross-linked with a trifunctional imine. C-55A propellant delivers a specific impulse (I_{sp}) representative of state-of-the-art aluminum-ammonium perchlorate (Al-AP) composite propellants. It has excellent physical properties and is ideal for use in case bonded motors. C-55A propellant will withstand prolonged storage at temperatures up to 135°F with little change in physical properties if the propellant is protected by a moisture seal. C-55A propellant is currently being used in a rocket motor developed by the U. S. Naval Ordnance Test Station (NOTS), China Lake, California designated NOTS Mod 401A rocket motor. (UNCLASSIFIED)

U. S. NAVAL ORDNANCE TEST STATION
China Lake, California
April 1966

U. S. NAVAL ORDNANCE TEST STATION

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FOREWORD

The purpose of this reported research was to study the ballistic and physical properties of C-55A, a cast composite propellant. The theoretical and practical aspects of the research were supported by experimentation.

This research was conducted under WepTask No. AWS-201-000/216-1/W002C at the U. S. Naval Ordnance Test Station (NOTS), China Lake, California. The information herein covers work that was completed December 1965.

This report has been reviewed for technical accuracy by H. L. Bennett and J. P. Diebold.

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INTRODUCTION

The C-55A cast composite propellant was developed by the U. S. Naval Ordnance Test Station (NOTS), China Lake, California, to support the Extended Range ASROC (ERA) program. As part of the ERA feasibility program, Code 457 developed the NOTS Mod 401A rocket motor. The C-55A propellant was tailored from an existing formulation for use in this motor.

To meet the requirements of the Mod 401A motor, it was necessary that the propellant have the following characteristics:

1. A cast composite capable of case bonding
2. Deliver 245 seconds specific impulse (I_{sp}) at 1,000 psi expanded to 14.7 psi with a 15 degree half angle
3. Be capable of operation between the temperature limits of 0 to 120°F and storage between -30 to 130°F. It must have a service life requirement of 5 years minimum
4. Have a burning rate near 0.5 in/sec at 1,000 psi
5. Have a π_K (percent change burn rate per °F at constant K) of 0.15 or less
6. Propellant must be Class B Interstate Commerce Commision (ICC)
7. Must have an autoignition temperature or 250°F or higher

In addition to the above operational requirements, other developmental aspects were considered equally important. This refers to the work that must be done to assure that the propellant is an end product ready for production. In addition to fully characterizing the ballistic and physical properties of the propellant, determination of the effects of processing variations on the finished propellant must be made. Sufficient knowledge must be available to qualify raw material lots for production. Since raw materials may change from lot-to-lot, sufficient knowledge must be available to enable adjustment in formulation to assure that the required ballistic and physical properties will be maintained.

PROPELLANT CHARACTERISTICS

FORMULATION

The C-55A propellant utilizes a carboxyl-terminated-polybutadiene binder. The binder is cross-linked with a trifunctional imine (HX-868). The formulation for C-55A is as follows:

Constituent	Percent by weight	Function
Butarez CTL-Type II	13.074[a]	Binder
HX-868	0.426[a]	Cross-linker
Yellow iron oxide ($Fe_2O_3 \cdot H_2O$)	0.5	Burning rate modifier
Al H-5	17.0	Fuel
NH_4ClO_4(AP)	69.0	Oxidizer

[a]These values are approximate and may change with raw material lots.

The following is a detailed explanation of the constituents:

1. Butarez CTL-Type II[1] is a carboxyl-terminated-polybutadiene polymer, containing 1.65 to 1.75 percent by weight active COOH groups; it contains no plasticizer. The active COOH groups are located primarily at the ends of the long polybutadiene chain. The average molecular weight range of the polymer is 4,800 to 5,600.

2. HX-868[2], is a trifunctional imine (1-, 3-, 5-tris-(carboxyl-2-ethyl-1-aziridine) benzene). The structure of HX-868 is shown in the following illustration.

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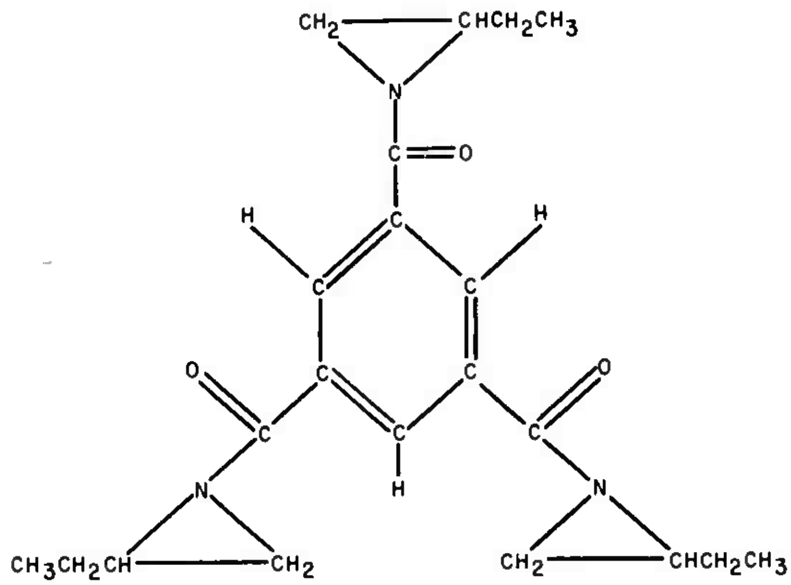
[1]Phillips Petroleum Company, Special Products Division, Bartlesville, Oklahoma.

[2]Minnesota Mining and Manufacturing Corporation (3M), St. Paul 1, Minnesota.



3. Yellow iron oxide[3] (Lemon 100) is a monohydrate of ferric oxide with the structural formula $Fe_2O_3 \cdot H_2O$.

4. Aluminum powder, Al H-5[4] has an average particle size of approximately 5 microns.

5. Ammonium perchlorate (AP)[5], used in C-55A is a blend of three particle sizes. The blend consists of 25 percent ground, 50 percent ordnance grade, and 25 percent spherical AP, with average particule sizes of approximately 20, 180, and 600 microns, respectively.

More detailed information is presented on page 26 in this report.

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[3]Columbian Carbon Company, c/o Dougherty Company, Los Angeles, California.

[4]Valley Metallurgical Processing Company, Essex, Connecticut.

[5]Pacific Engineering Production Company, Henderson, Nevada, and American Potash and Chemical Company (AP&CC), Los Angeles, California.

BALLISTIC PROPERTIES

The burning rate (r_b) of C-55A propellant can be expressed as the equation $r_b = cP^n$. The following burning rate information was obtained by static firing 5-inch-diameter rocket motors at 0, 70, and 135°F over a pressure range from 200 to 2,000 psi. These motors contained 6.2 pounds of propellant and had a 1.5-inch web. The grain had a cylindrical perforation with the forward end inhibited and the aft end uninhibited. The theoretical burning surface area remained constant throughout the entire burn.

$r_b = 0.0281\ P^{0.40}$ valid from P = 600 to 2,000 psia at 70°F

$r_b = 0.0293\ P^{0.40}$ valid from P = 600 to 2,000 psia at 135°F

$r_b = 0.0268\ P^{0.40}$ valid from P = 600 to 2,000 psia at 0°F

$\pi_K = 0.12\%$ / °F for $\overline{P}$ = 1,000 psia from 0 to 135°F

$\pi_K = 0.12\%$ / °F for $\overline{P}$ = 700 psia from 0 to 135°F

$\pi_K = 0.12\%$ / °F for $\overline{P}$ = 1,500 psia from 0 to 135°F

$\pi_{P/T} = 0.12\%$ / °F for $\overline{P}$ = 1,000 psia from 0 to 135°F

$\pi_{P/T} = 0.12\%$ / °F for $\overline{P}$ = 700 psia from 0 to 135°F

$\sigma_p = 0.069\%$ / °F at P = 1,000 psia from 0 to 135°F

$\sigma_p = 0.069\%$ / °F at P = 700 psia from 0 to 135°F

where:

π_K = Temperature coefficient of pressure at constant K_n

$\pi_{P/T}$ = Temperature coefficient of burning rate at constant P/T

σ_p = Temperature coefficient of burning rate at constant P

Figure 1 shows the burning rate versus pressure for C-55A propellant conditioned to 70°F. The r_b line levels slightly at the lower pressures. To characterize C-55A propellant, most of the 5-inch motors were fired at pressures from 600 to 2,000 psi. Only a limited number of motors were fired at the lower pressures. However, both the motor firings and strand burning data indicate that the burning rate line does curve at the lower pressures.

Figure 1 also shows a K_n versus P curve for C-55A propellant. The K_n curve was calculated from the r_b curve. To calculate K_n, the following equation was used:

$$P = \frac{S_b \, r_b \rho}{C_d \, A_t}$$

since

$$K_n = \frac{S_b}{A_t}$$

$$K_n = \frac{P \, C_d}{r_b \rho}$$

but $r_b = 0.0281 \, P^{0.40}$ at 70°F,

therefore,

$$K_n = \frac{C_d P^{0.60}}{\rho 0.0281} = 3.41 \, P^{0.60} \text{ at } 70°F$$

valid from P = 600 to 2,000 psi

where:

P = Pressure (psi)

S_b = Burning surface area (in^2)

r_b = Burning rate (in/sec)

ρ = Density (lb/in^3)

C_d = Discharge coefficient (1/sec)

A_t = Throat area (in^2)

K_n = Ratio of burning surface to area of throat

The value of C_d used in this equation is 0.006 1/sec. This value of C_d was obtained from the firings of the NOTS Mod 401A motor.

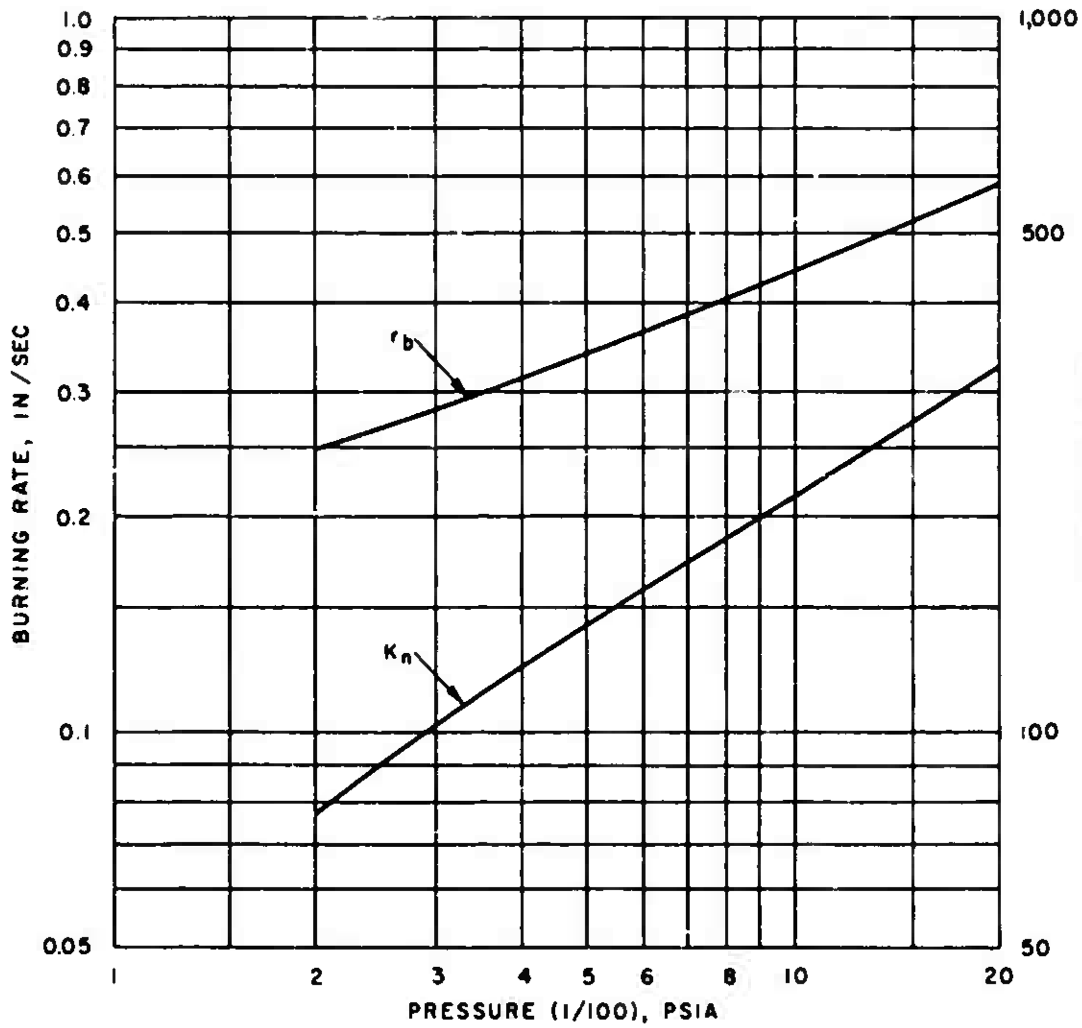

FIG. 1. Burning Rate and K_n Versus Pressure for C-55A Propellant Conditioned tc 70°F.

C-55A has delivered an I_{sp} of 245 seconds at an average pressure of 1,050 psi with a nozzle expansion ratio of 8.5:1. This was shown in the NOTS Mod 401A motor which contains 415 pounds of propellant. C-55A propellant has performed as designed through 14 static tests and 6 flight tests of the NOTS Mod 401A motor. Other ballistic and thermodynamic properties are listed in Table 1.

Table 2 gives the empirical formula and the combustion products in both the chamber and exit equilibrium condition. These calculations were performed with 0.5-percent $Fe_2O_3 \cdot H_2O$ replaced by 0.5 percent NH_4ClO_4.

TABLE 1. Ballistic and Thermodynamic Properties

I_{sp} (1,000/14.7) theoretical, frozen composition	257 sec
I_{sp} (1,000/14.7) theoretical, shifting composition	266 sec
C* (characteristic exhaust velocity)	5,173 ft/sec
C* (measured)	5,220 ft/sec
Heat of explosion (measured)	1,545 cal/g
Flame temperature in chamber 1,000 psia (equilibrium composition)	5807°F
Flame temperature exit conditions, 14.7 psia (equilibrium composition)	3409°F
Mean molecular weight of products, in chamber	25.70 g/g mole
Mean molecular weight of products, equilibrium exit condition	26.35 g/g mole
Mean molecular weight of gases, in chamber	19.40 g/g mole
Mean molecular weight of gases, equilibrium exit condition	19.52 g/g mole
Combustion gas specific heat ratio γ, in chamber	1.191
Combustion gas specific heat ratio γ, exit condition	1.200

TABLE 2. Combustion Products in g-moles/100 g

Component	Chamber	Exit equil.	Component	Chamber	Exit equil.
Al	0.0009	...	CO	0.9264	0.9109
AlH	0.0002	...	N_2	0.2948	0.2957
OH	0.0257	0.0004	Al_2O_3 (C)[a]	......	0.3150
O_2	0.0003	...	$AlCl_3$	0.0014	...
AlCl	0.0303	...	AlO	0.0004	...
H_2	1.1254	1.2013	CO_2	0.0455	0.0610
AlOCl	0.0001	...	HCl	0.5143	0.5882
AlO_2H	0.0025	...	NH	0.0001	0.0000
Cl	0.0423	0.0031	O	0.0018	...
H	0.1378	0.0086	Al_2O_3 (L)[b]	0.2971	...
H_2O	0.4441	0.4100	Cl_2	0.0001	...
			NO	0.0018	...

Empirical Formula (g-atoms/100 g)

Carbon	0.97194	Nitrogen	0.59150
Hydrogen	3.81972	Aluminum	0.63009
Oxygen	2.38841	Chlorine	0.59150

[a]C = crystalline

[b]L = liquid

PHYSICAL PROPERTIES

Table 3 shows the tensile properties of C-55A at different strain rates and temperatures. Figure 2 is a plot of tensile strength versus strain rate at different temperatures. The tests were performed on a table model Instron machine using a JANAF CPS-1 sample. The samples were prepared by casting and curing the propellant in a 3- x 5- x 7-inch waxed carton. Then the propellant was machined into 0.400-inch slabs; the tensile test specimens were cut from these slabs.

TABLE 3. Tensile Properties of C-55A

Temp., °F	Crosshead speed, in/min	Maximum tensile, psi	Elongation maximum tensile, %	Elongation rupture, %	10% Modulus, psi
-50	0.02	180	38	44	Initial 1,918
-50	0.2	223	33	35	Initial 3,598
-50	2.0	324	26	32	5,374
0	0.02	98	57	57	744
0	0.2	127	70	70	662
0	2.0	162	59	59	1,101
77	0.2	77	52	59	295
77	2.0	98	57	61	406
77	20.0	129	57	57	746
130	0.2	57	62	62	220
130	2.0	72	72	72	288
130	20.0	94	75	75	403
180	0.2	49	35	35	203
180	2.0	61	44	44	246
180	20.0	78	70	70	319

Additional physical properties of C-55A propellant are listed below:

Density 77°F	0.0637 lb/in^3
Coefficient of linear thermal expansion 50 to 70°F	5.15×10^{-5} in/in-°F
Thermal conductivity 77 to 180°F	0.19 Btu/(hr) (ft) (°F)
Second order transition temperature	-102 ± 4°F
Shear strength at 77°F	890 psi

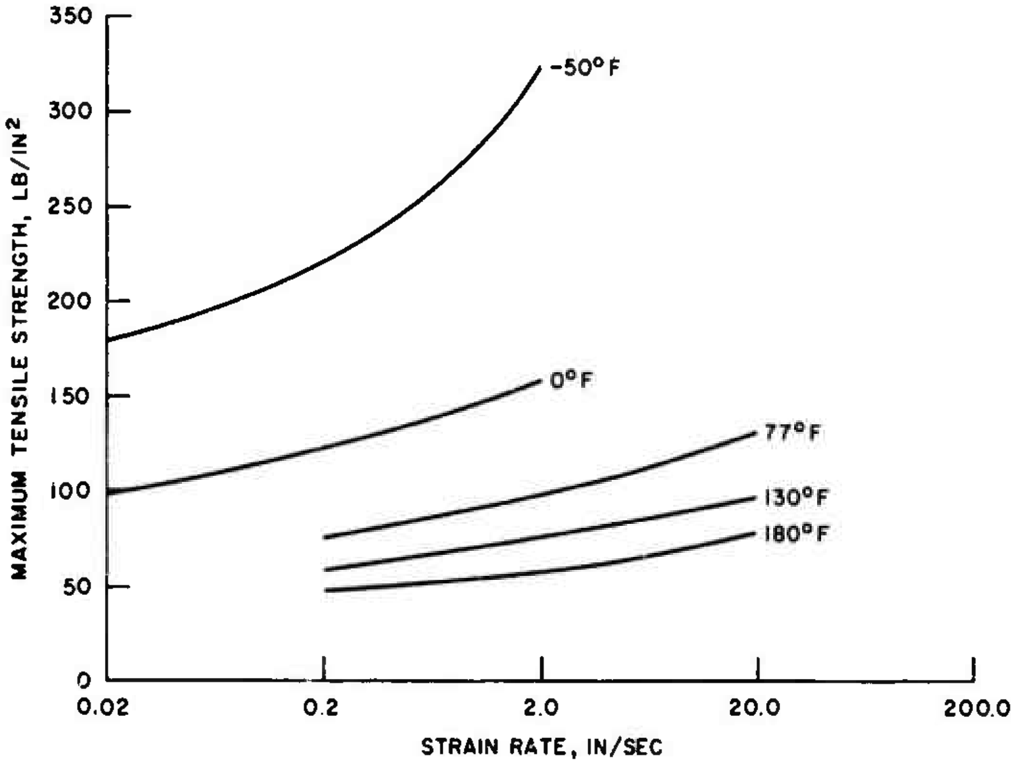

FIG. 2. Maximum Tensile Strength Versus Strain Rate at Various Temperatures.

The ASTM 732-46 shear strength test was used with the following exceptions: (1) 0.528-inch punch, (2) a 0.2-in/min crosshead speed, and (3) the sample was not supported at the bottom of the punch.

SENSITIVITY AND THERMAL STABILITY

A card gap test was performed on C-55A propellant and no detonation occurred at zero cards. This test consisted of casting 2-inch-diameter test samples in pipes 5 1/2 inches in length. The propellant, when cured, bonded to the pipe. A donor charge of high explosive was placed on the sample and a witness plate placed under the sample. After the donor charge was detonated, the witness plate showed that no detonation of the propellant sample had occurred.

Impact sensitivity tests were conducted using a 2 kg weight with a 35-mg 0.033-inch-thick slice of propellant placed on 50-180 grit sandpaper.[6] The 50 percent fire point was 11 cm. The no fire point was 9 cm. The electrostatic sensitivity test gave no fires at 12.5 joules (maximum capacity of the machine). The Allegany Ballistics Laboratory (ABL) friction test gave a zero initiation level of 60 to 100 pounds (depending on the batch tested) at 77°F. The propellant has been given an ICC Class B rating.

Other tests performed on C-55A propellant include: (1) differential thermal analysis (DTA), (2) gas profile analysis (GPA), and (3) isothermal analysis. The DTA test consists of heating a small 15- to 25-mg sample at specified rates of 5 to 25°F/min, and measuring the endothermic and exothermic peaks that occur. The onset temperature of the peaks and the slope of the curves can be used for a direct indication of thermal stability, since the first exotherm usually is the first evidence that thermal decomposition is taking place.

The gas profile analysis consists of passing a carrier gas (helium) through a 100-mg sample (mixed with 0.1-mm glass beads) while it is being heated at approximately 11°F/min. A detector measures any gases emitted from the propellant. The temperature at which the first gas is given off is a good indication of thermal stability. For C-55A this GPA temperature is approximately 365°F. This corresponds very closely to the first DTA exotherm of 390°F.

Isothermal analysis (cook-off) was made on 1-, 2-, and 5-inch-diameter samples, and the data is given in Table 4.

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[6] Carborundum Co., Niagara Falls, New York

TABLE 4. C-55A Cook-Off Time

Temp., °F	5-in. diam., hr	2-in. diam., hr	1-in. diam., hr
320	no cook-off after 168	...	...
335	33.4	...	...
350	14.6	...	...
360	...	26.3	...
375	...	4.5	16.7
388	...	...	20.0

These data along with DTA data, were used to calculate the critical temperature for C-55A propellant. The critical temperature is defined as the maximum surface temperature that will allow a steady state temperature distribution in a mass of propellant such as a grain. Critical temperatures for cylinders and spheres of various diameters are given in Table 5. See NAVWEPS Report 8388 for explanation of critical temperature calculations.

TABLE 5. Theoretical Critical Temperatures for C-55A Propellant Samples

Cylinders		Spheres	
Diam., in.	Critical temp., °F	Diam., in.	Critical temp., °F
1	362	1	371
2	337	2	346
5	306	5	314
12	278	12	286

PROPELLANT AGING

Aging studies were conducted under the following conditions:

1. Propellant slabs, 0.400-inch thick were aged at 70, 135, and 180°F dry and 79 percent relative humidity.

2. Propellant samples were aged in 3- x 4- x 5-inch waxed cartons at 70, 135, and 180°F dry and 79 percent relative humidity.

At predetermined time intervals, the samples were removed from the conditioning environment and tested. The propellant samples that were aged in waxed cartons were machined into 0.400-inch slabs, and then cut into tensile test specimens. The test specimens were then pulled at 77°F on the Instron tensile tester at 2.0 in/min crosshead speed. The samples were machined as shown in Fig. 3.

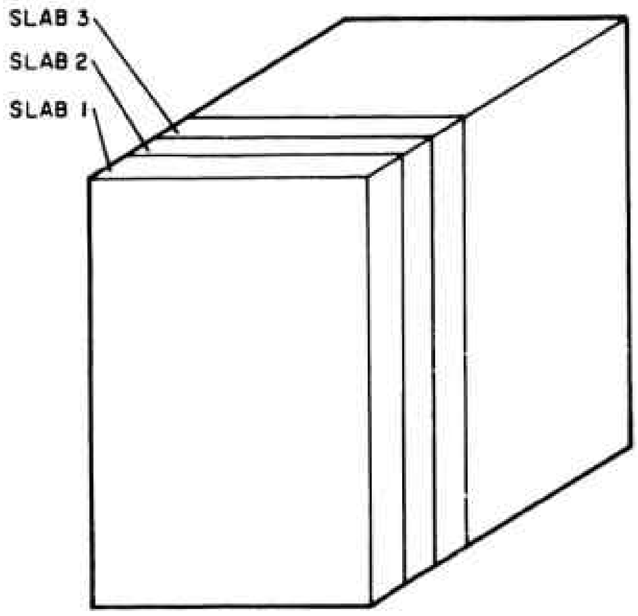

FIG. 3. Propellant Test Samples.

Note that only the outside surface of Slab 1 was exposed to atmosphere during aging. This is important, for the data show that there is a great deal of atmospheric effect on the outer slab.

Tables 6 through 8 tabulate the tensile properties measured on the aged 0.400-inch slabs. Slab aging is not necessarily characteristic of the process which occurs in a rocket motor because the slabs are relatively thin and are exposed to atmosphere on all sides. The tensile properties of the aged slabs are due primarily to skin effects. This is evident when the tensile properties of the first slab cut from the aged carton are compared with the tensile properties of the second and third slab. Values given in tables 6 through 11 are an average of two or three test samples.

TABLE 6. Aging of C-55A Propellant at 70°F
(0.400-inch-thick slabs)

	Dry				79% Relative humidity			
Aging time, days	S_m, psi	E_m, %	E_r, %	Mod, psi	S_m, psi	E_m, %	E_r, %	Mod, psi
Initial	81	56	69	379	81	56	69	379
1	80	54	59	389	72	45	53	312
2	84	61	65	384	69	42	51	338
4	89	57	59	355	65	38	47	334
7	87	59	65	374	63	34	46	367
14	94	52	59	450	57	33	48	327
30	101	50	60	409	55	28	33	393
60	99	45	49	488	50	30	40	335
120	101	44	50	470	42	22	34	360

S_m - Maximum tensile strength, psi

E_m - Elongation at maximum tensile strength, percent

E_r - Elongation at rupture, percent

Mod - Modulus, psi

TABLE 7. Aging of C-55A Propellant at 135°F
(0.400-inch-thick slabs)

	Dry				79% Relative humidity			
Aging time, days	S_m, psi	E_m, %	E_r, %	Mod, psi	S_m, psi	E_m, %	E_r, %	Mod, psi
Initial	81	56	69	379	81	56	69	379
1	78	53	60	328	51	29	39	332
2	82	52	56	430	42	26	35	313
4	90	55	58	397	38	22	30	319
7	90	56	61	452	34	26	34	274
14	99	44	46	482	30	18	21	273
30	114	40	44	648	34	19	22	409
60	127	24	24	979	42	13	13	546
120	145	14	15	1,940	62	4	6	2,260

S_m - Maximum tensile strength, psi

E_m - Elongation at maximum tensile strength, percent

E_r - Elongation at rupture, percent

Mod - Modulus, psi

TABLE 8. Aging of C-55A Propellant at 180°F
(0.400-inch-thick slabs)

	Dry				79% Relative humidity			
Aging time, days	S_m, psi	E_m, %	E_r, %	Mod, psi	S_m, psi	E_m, %	E_r, %	Mod, psi
Initial	81	56	69	379	81	56	69	379
1	76	48	56	370	63	37	49	324
2	77	50	54	359	56	26	36	374
4	82	44	46	400	43	27	37	320
7	84	41	43	420	38	27	38	309
14	94	26	26	667	39	23	24	338
30	98	12	13	1,345	47	19	19	673
60	94	5	5	2,770	52	9	9	1,527
120	86	3	4	5,200	68	5	6	3,400

S_m - Maximum tensile strength, psi

E_m - Elongation at maximum tensile strength, percent

E_r - Elongation at rupture, percent

Mod - Modulus, psi

Tables 9 through 11 contain data obtained from the cartons of aged propellant. It is important to note the difference between Slabs 1, 2, and 3 cut from the same carton. This comparison emphasizes the extreme skin effects caused by exposure to atmosphere. It is also interesting to note how rapidly these effects disappear with depth. Slab 1 is only 0.400-inch thick, but the skin effects have already become negligible in Slab 2.

Figures 4 through 9 contain a graphic presentation of the maximum tensile strength and elongation (at maximum tensile) data obtained from the aged cartons. Remember, Slabs 1, 2, and 3 refer to the order in which they were machined off the carton, Slab 1 being on the end and exposed to atmosphere. The initial point on the graphs (0 day aged) is the condition of the propellant after curing 3 days at 135°F.

TABLE 9. Aging of C-55A Propellant at 70°F (cartons)

		Dry				79% relative humidity			
Aging time, days	Slab no.	S_m, psi	E_m, %	E_r, %	Mod, psi	S_m, psi	E_m, %	E_r, %	Mod, psi
	Initial	81	56	69	379	81	56	69	379
	1	100	54	58	397	72	39	44	377
30	2	92	59	60	333	88	56	64	353
	3	91	58	60	346	91	59	61	347
	1	98	48	50	433	69	35	39	375
60	2	92	58	64	364	85	49	51	351
	3	90	63	68	326	83	53	62	362
	1	100	42	45	495	72	29	32	515
120	2	94	48	54	435	79	44	48	450
	3	90	50	54	435	78	46	58	435

S_m - Maximum tensile strength, psi
E_m - Elongation at maximum tensile strength, percent
E_r - Elongation at rupture, percent
Mod - Modulus, psi

TABLE 10. Aging of C-55A Propellant at 135°F (cartons)

		Dry				79% relative humidity			
Aging time, days	Slab no.	S_m, psi	E_m, %	E_r, %	Mod psi	S_m, psi	E_m, %	E_r, %	Mod, psi
	Initial	81	56	69	379	81	56	69	379
	1	100	41	43	491	33	19	21	364
30	2	87	58	62	311	59	43	53	264
	3	86	59	63	308	68	52	57	250
	1	117	16	18	1,260	17	8	8	240
120	2	91	56	60	410	29	21	23	275
	3	90	57	65	420	38	29	34	225

S_m - Maximum tensile strength, psi

E_m - Elongation at maximum tensile strength, percent

E_r - Elongation at rupture, percent

Mod - Modulus, psi

TABLE 11. Aging of C-55A Propellant at 180°F (cartons)

		Dry				79% relative humidity			
Aging time, days	Slab no.	S_m, psi	E_m, %	E_r, %	Mod, psi	S_m, psi	E_m, %	E_r, %	Mod, psi
	Initial	81	56	69	379	81	56	69	179
	1	55	13	13	642	37	14	15	515
30	2	55	57	59	151	47	36	42	219
	3	59	59	62	163	50	43	53	203
	1	68	3	5	2,930	44	14	16	645
120	2	34	44	51	135	41	15	16	530
	3	32	48	55	120	41	16	17	505

S_m - Maximum tensile strength, psi

E_m - Elongation at maximum tensile strength, percent

E_r - Elongation at rupture, percent

Mod - Modulus, psi

An examination of the graphs reveals several things that are readily apparent. First, Slab 1 always shows a pronounced difference from Slabs 2 and 3. In all three dry, aged conditions, Slab 1 has a noticeably higher tensile strength and a lower elongation. Also, in the wet aging condition, Slab 1 has always degraded to a much greater extent than Slabs 2 and 3. This shows that regardless of whether it is moisture or some other ingredient in the atmosphere that affects the propellant, there is definitely a marked skin-effect. It should also be noted that this effect decreases quite rapidly with depth. This skin-effect is the reason that the aging of propellant slabs is not deemed very useful. It is felt that the slabs will exhibit nearly all skin-effect and this is not representative of what happens in a rocket motor.

Moisture has a very detrimental effect on the propellant at all temperatures. A rocket motor using C-55A propellant should be sealed against atmospheric moisture. The data shows, that under dry conditions, the propellant retains good physical properties even at 135°F. Under wet conditions, the propellant will degrade at room temperature.

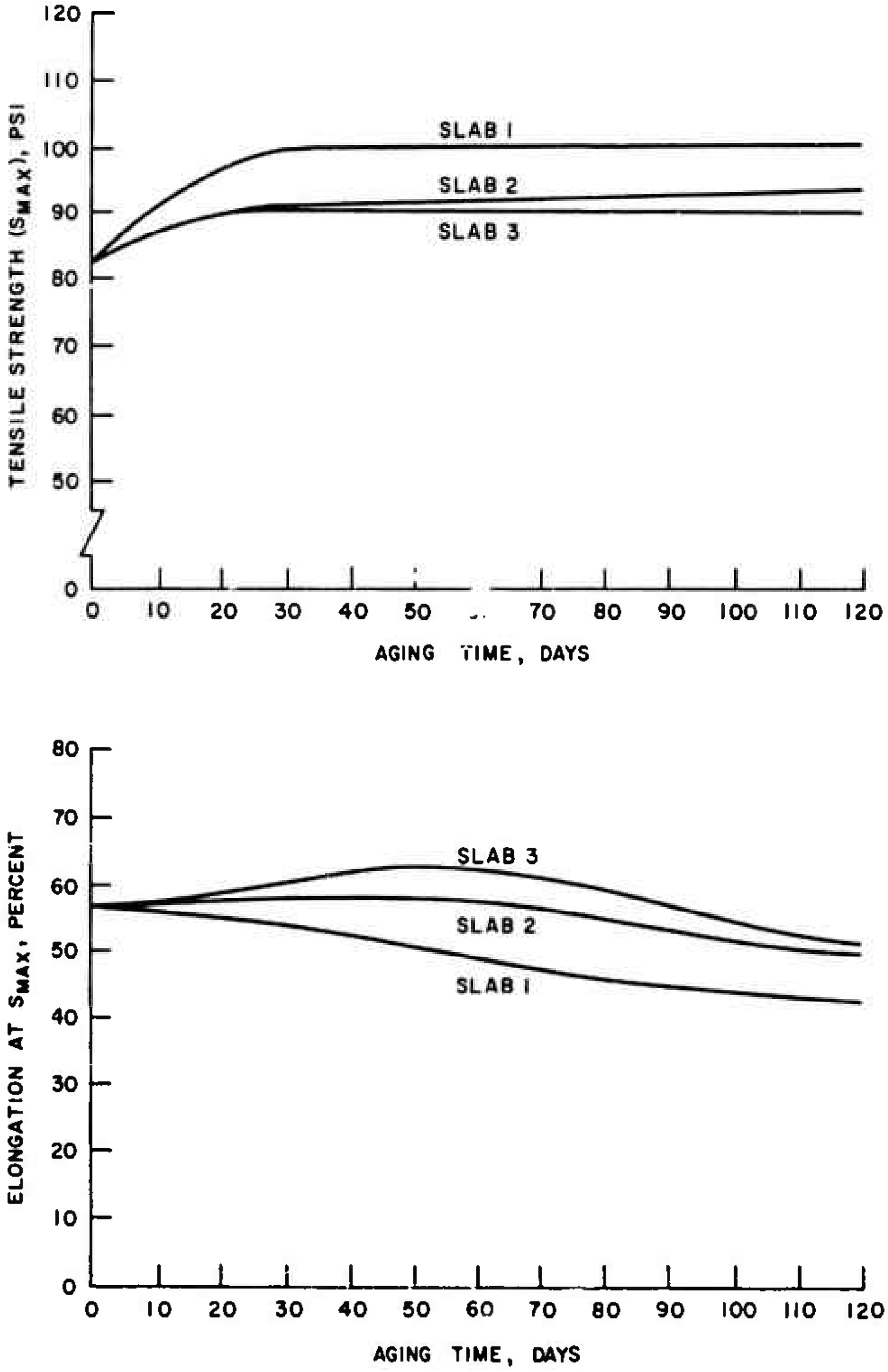

FIG. 4. Tensile Strength and Elongation After Aging at 77°F Dry.

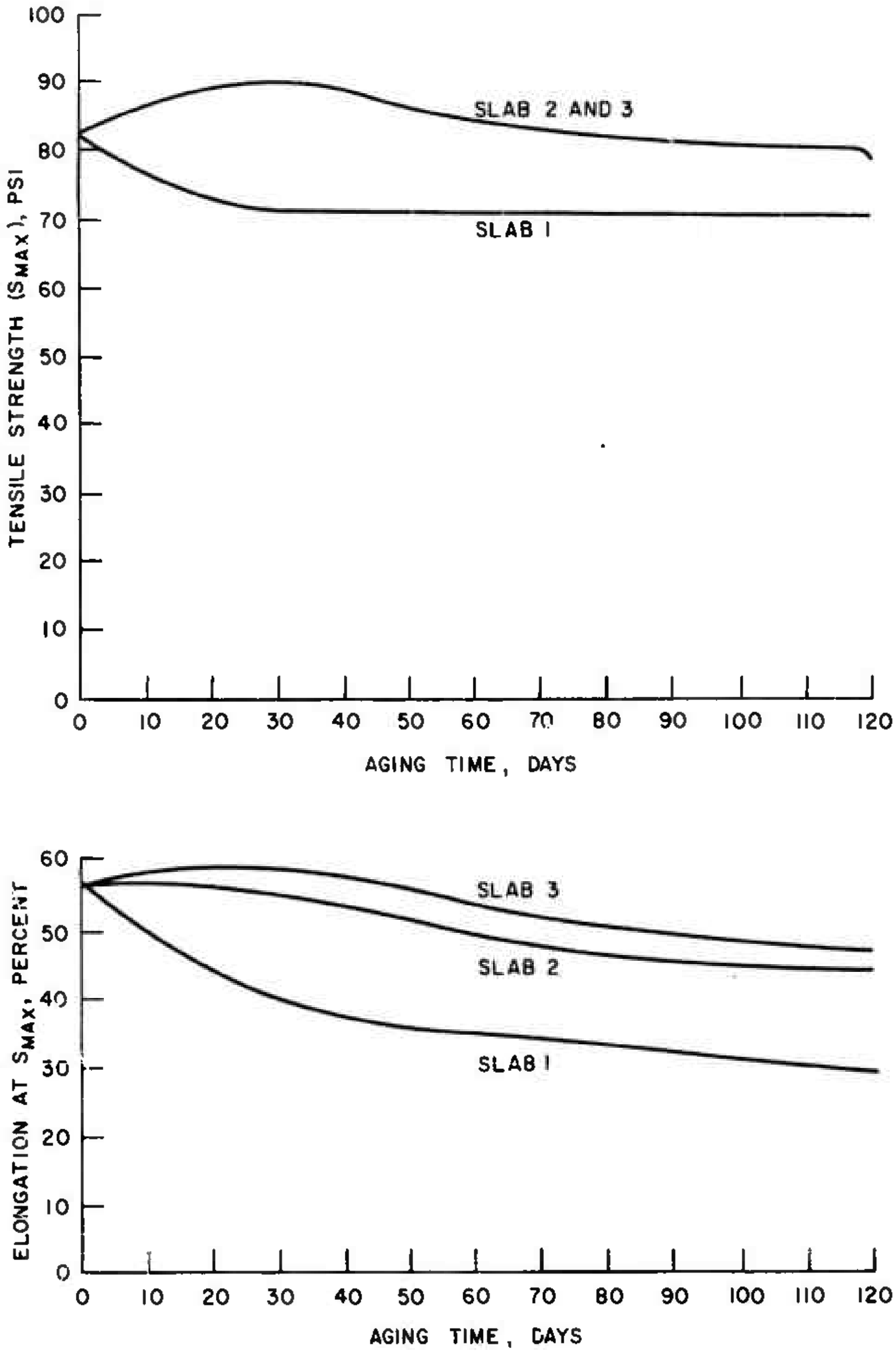

FIG. 5. Tensile Strength and Elongation After Aging at 77°F and 79 Percent Relative Humidity.

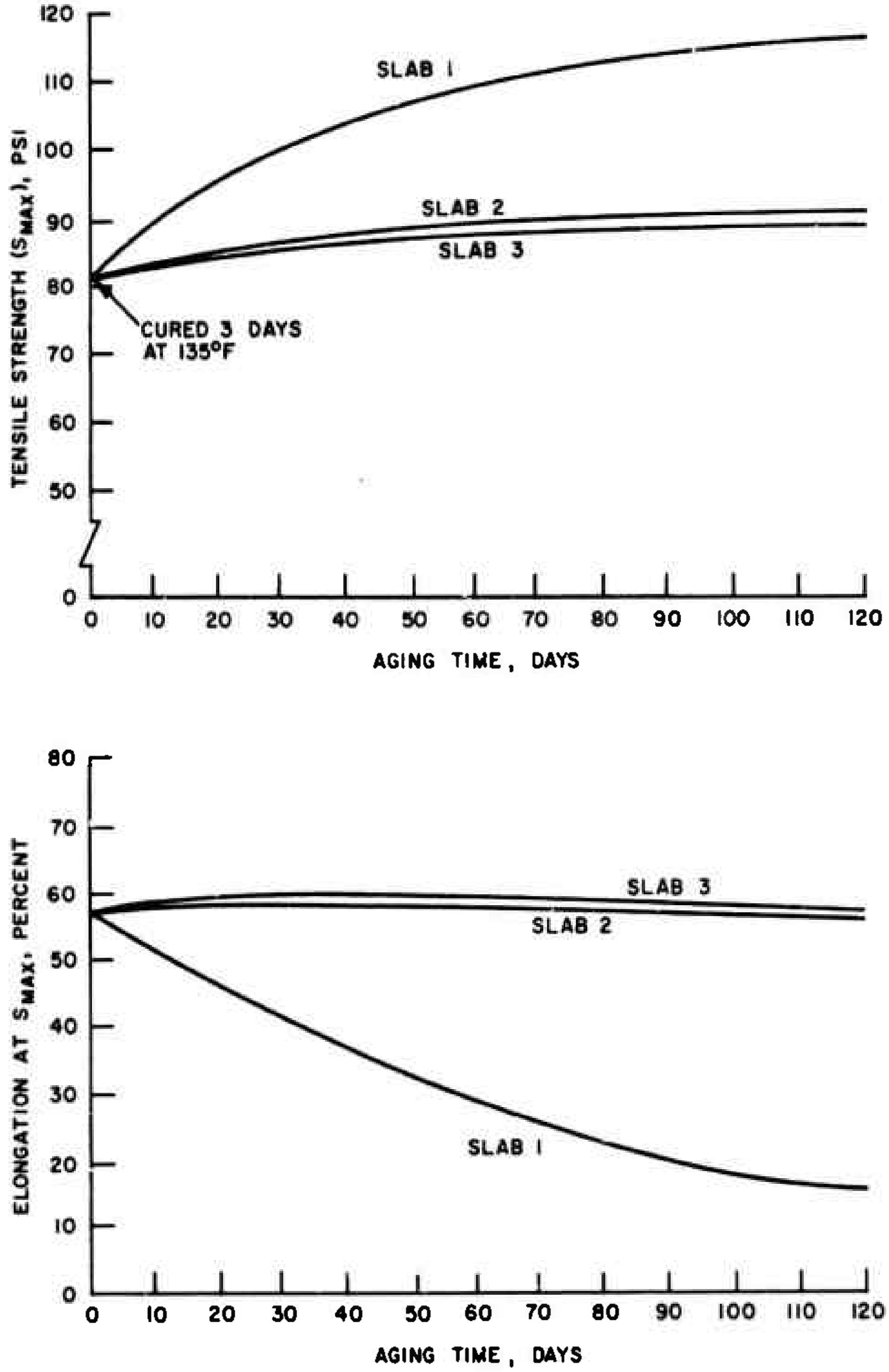

FIG. 6. **Tensile Strength and Elongation After Aging at 135°F Ambient Relative Humidity. (Less Than 2% R. H.)**

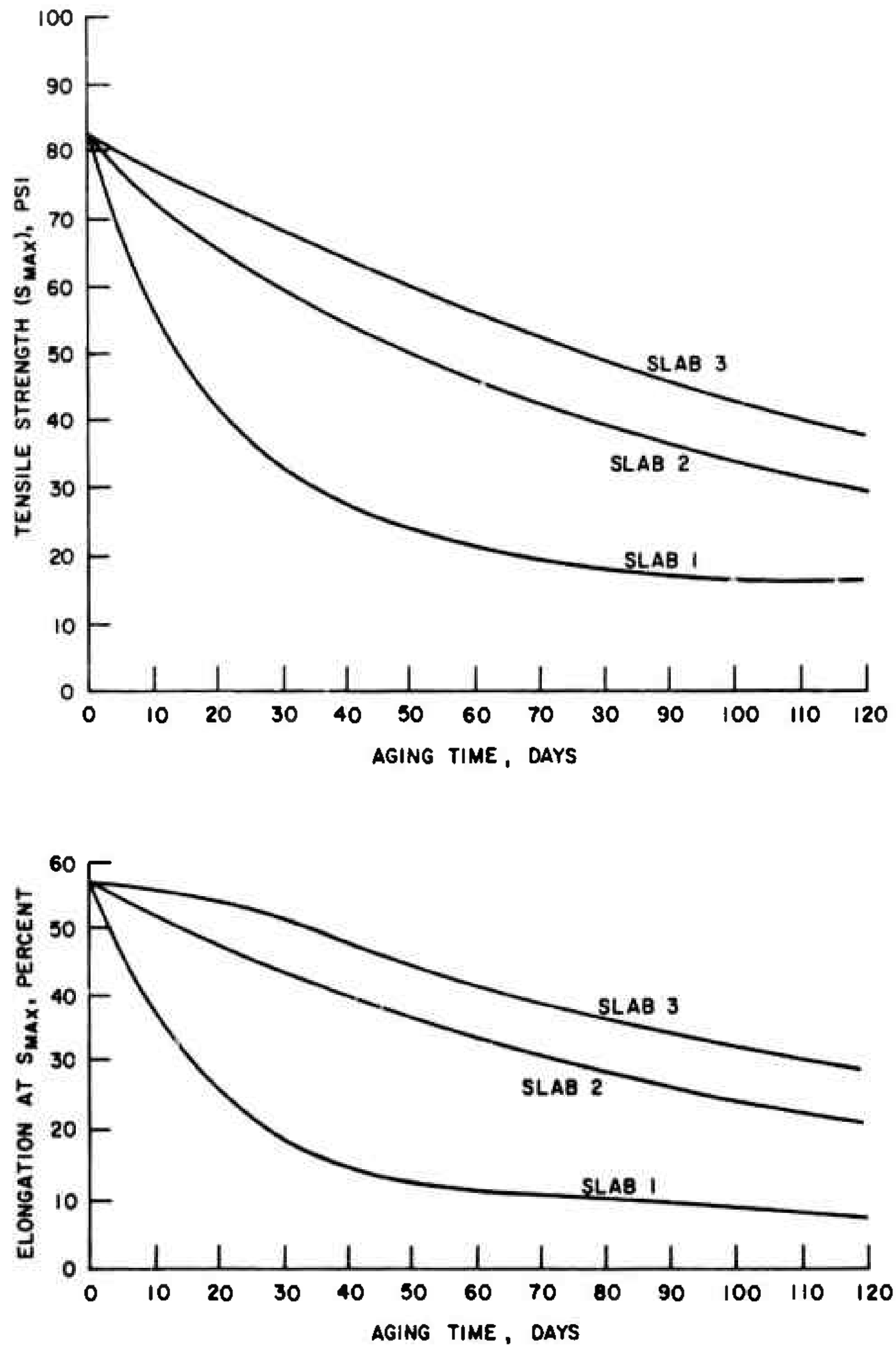

FIG. 7. Tensile Strength and Elongation After Aging at 135°F and 79 Percent Relative Humidity.

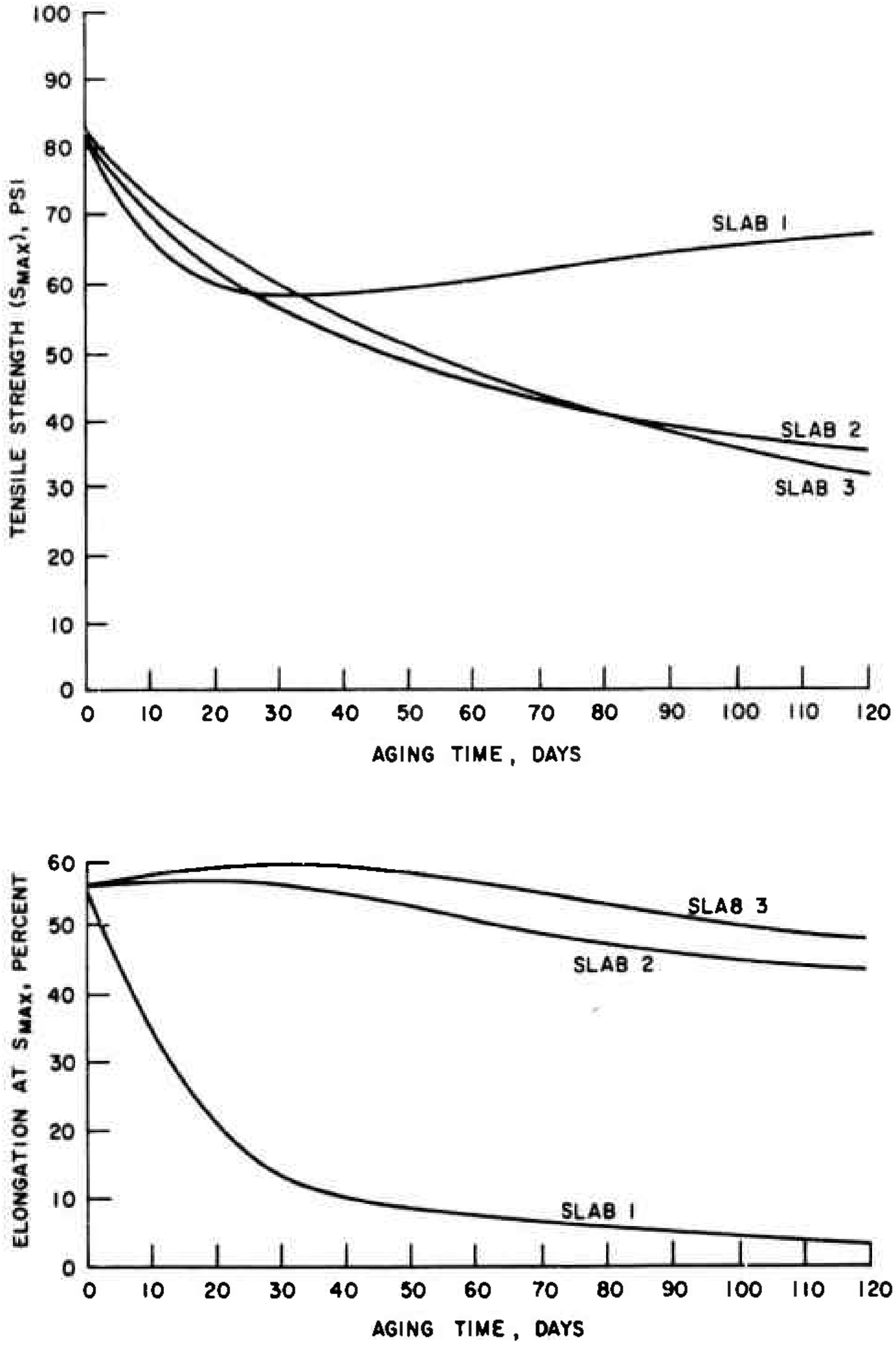

FIG. 8. Tensile Strength and Elongation After Aging at Ambient Relative Humidity of Less Than 2% at 180°F.

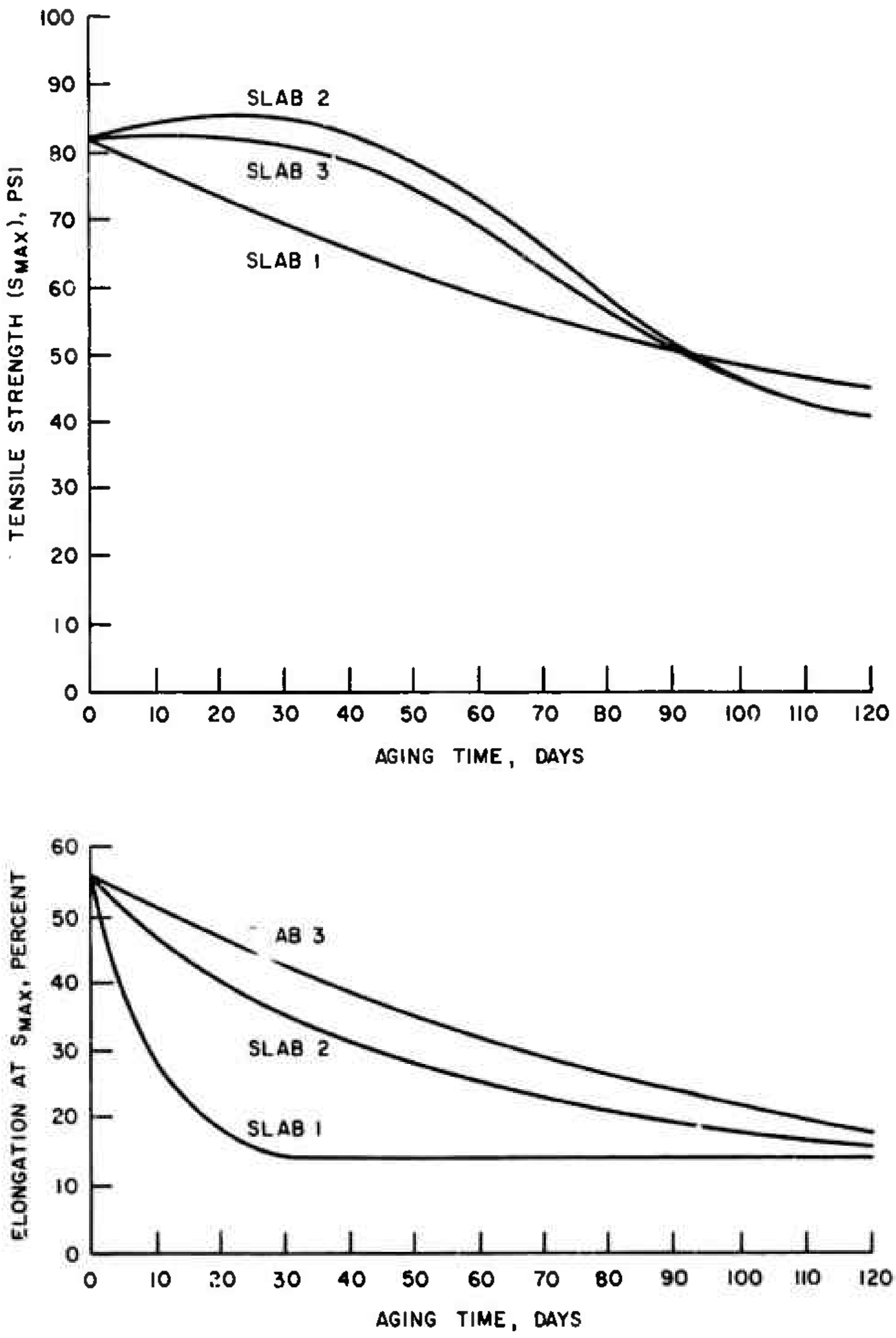

FIG. 9. Tensile Strength and Elongation After Aging at 180°F and 79 Percent Relative Humidity.

The data also show an unexpected occurrence at the 180°F low relative humidity condition. Judging from the data taken at 77 and 135°F dry, one would expect the propellant to become harder (i. e., higher tensile-lower elongation) at the higher temperature of 180°F. This is not what happened. Instead, the propellant lost tensile strength quite rapidly and also exhibited a low elongation (quite marked in Slab 1). No explanation will be offered for this phenomenon; it will only be pointed out that at 180°F a general degradation takes place even under dry conditions. This is a definite indication that reactions occur at 180°F that differ from the reactions at the lower temperatures, or at least a different reaction dominates at the higher temperature.

RAW MATERIAL PREPARATION AND QUALITY CONTROL

OXIDIZER

The oxidizer used in C-55A is a blend of three particle size regions of crystalline AP; 25-percent large spheroidal, 50-percent ordnance grade, and 25-percent ground AP (ground at NOTS) with average particle sizes of 600, 180, and 20 microns, respectively. (See Appendix A for specifications.)

The ground AP is made from the AP & CC ordnance grade AP (180 micron region), ground in a Mikro Pulverizer, Model 1-SH[7] at a hammer speed of 10,000 rpm, a feed speed of 88 rpm, and using a 0.020-inch herringbone screen.

Quality control of the AP is concerned primarily with maintaining the proper particle size distribution. To accomplish this, the individual ingredients are analyzed as they are received from the manufacturer, then after the grinding operation, and again after final blending in a twin shell blender. The analysis consists of determining the particle size distribution of the material by using Tyler[8] screens for the larger particle sizes, and a micromerograph and Fisher[9] subsieve sizer for smaller particle sizes. Micromerograph methods have given results that change with the elapsed time between grinding and the analysis operations.

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[7] Mikro Corporation, Summit, New Jersey

[8] W. S. Tyler Co., Cleveland, Ohio

[9] Fisher Instrument Manufacturing Division, Indiana, Pennsylvania

The AP particle size distribution is the mechanism by which the burning rate of C-55A propellant will be controlled in production. This means that the percentage of 25 to 50 to 25 for the three different particle size regions is not fixed, but may be changed as necessary for the propellant to meet burning rate specifications. Figure 10 shows a typical particle size distribution of the blend used in C-55A.

Several mixes of C-55A have been made with the oxidizer blend purposely varied. As expected, the blends with an increased percentage of ground AP oxidizer produced propellants with an increased burning rate, while blends which were low in ground AP oxidizer produced propellants with slower than normal burning rates (Fig. 11). This method of controlling burning rate by varying the percentage of fine particle AP is standard throughout the propellant industry. In the production of a propellant such as C-55A, the ratios of ground, ordnance grade, and spheroidal AP may vary slightly because each lot of raw materials may vary.

BINDER PREPOLYMER AND CROSSLINKER

The binder prepolymer, Butarez CTL-Type II, is a carboxyl-terminated-polybutadiene polymer ranging from 1.65 to 1.75 percent by weight active COOH group. The active COOH groups are located primarily at the ends of the polybutadiene chain. Butarez CTL-Type II contains no plasticizer. The preliminary quality control of the prepolymer is a quantitative analysis for the COOH groups. This analysis is checked against the analysis supplied by the manufacturer. Based on these analyses, several 1-gallon mixes of C-55A are made in which the imine-to-carboxyl ratio is varied. These mixes are cured and the physical properties compared. The ratio producing the desired physical properties will be used with the given Butarez lot.

The crosslinker HX-868 (see page 2 of this report for the structural formula) is a trifunctional imine with a theoretical molecular weight of 369.47.

With respect to a change in purity, the HX-868 changes quite noticeably in appearance and reactivity. For example, a lot of HX-868 with an equivalent weight of 152 (81 percent pure) is a dark viscous liquid at room temperature Propellant batches made with this material would have to cure 10 days at 135°F at an imine-to-carboxyl ratio of 0.85 to achieve the desired physical properties. A lot of HX-868 with an equivalent weight of 136 (90.5 percent pure) is a cream-colored solid at room temperature. Propellant batches made with this material will have the desired physical properties after curing only 3 days at 135°F with an imine-to-carboxyl ratio as low as 0.625. HX-868 is probably a white crystalline solid in its pure state; however, 91 percent pure is the best quality that has been received at NOTS.

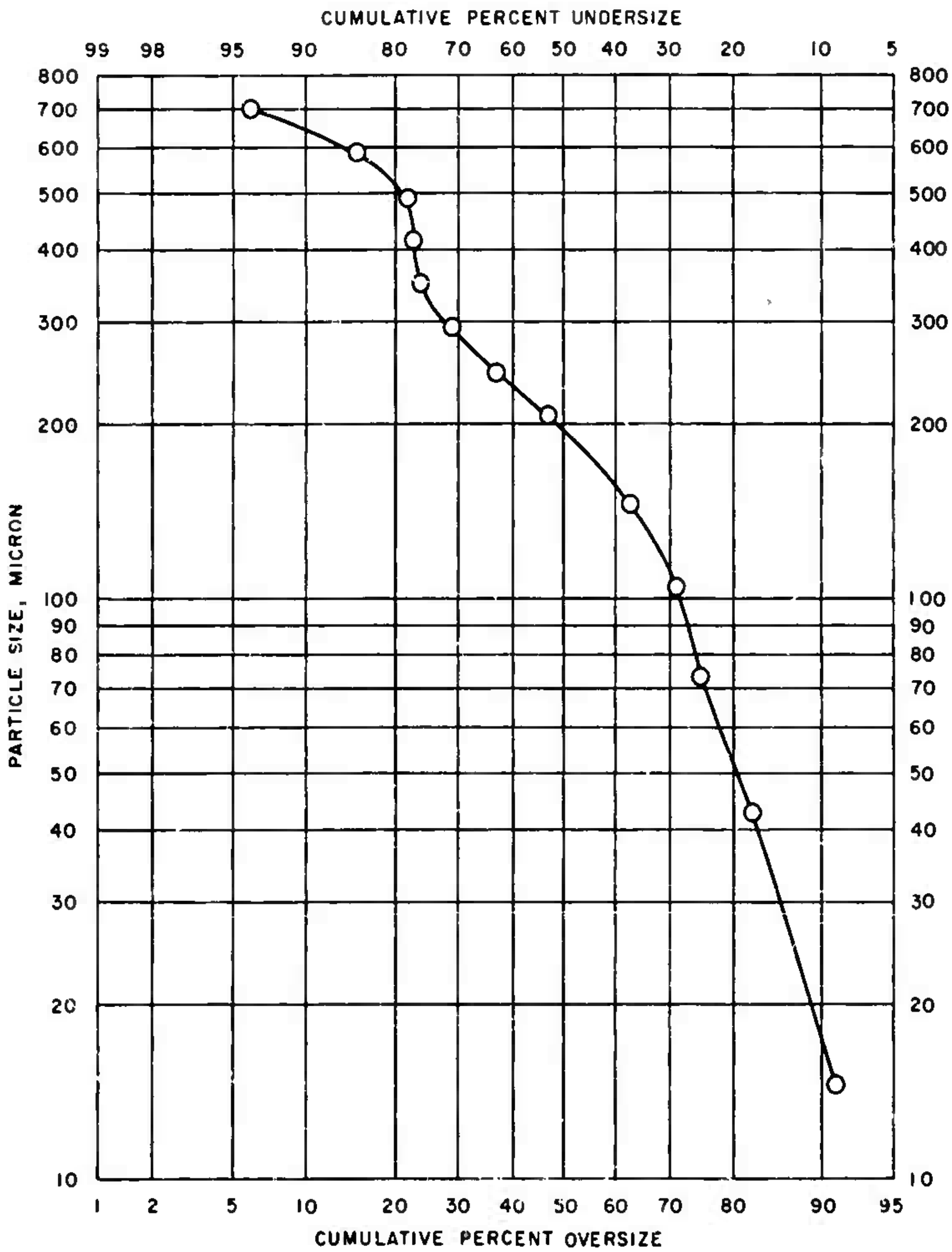

FIG. 0. Typical AP Particle Size Distribution for C-55A Propellant.

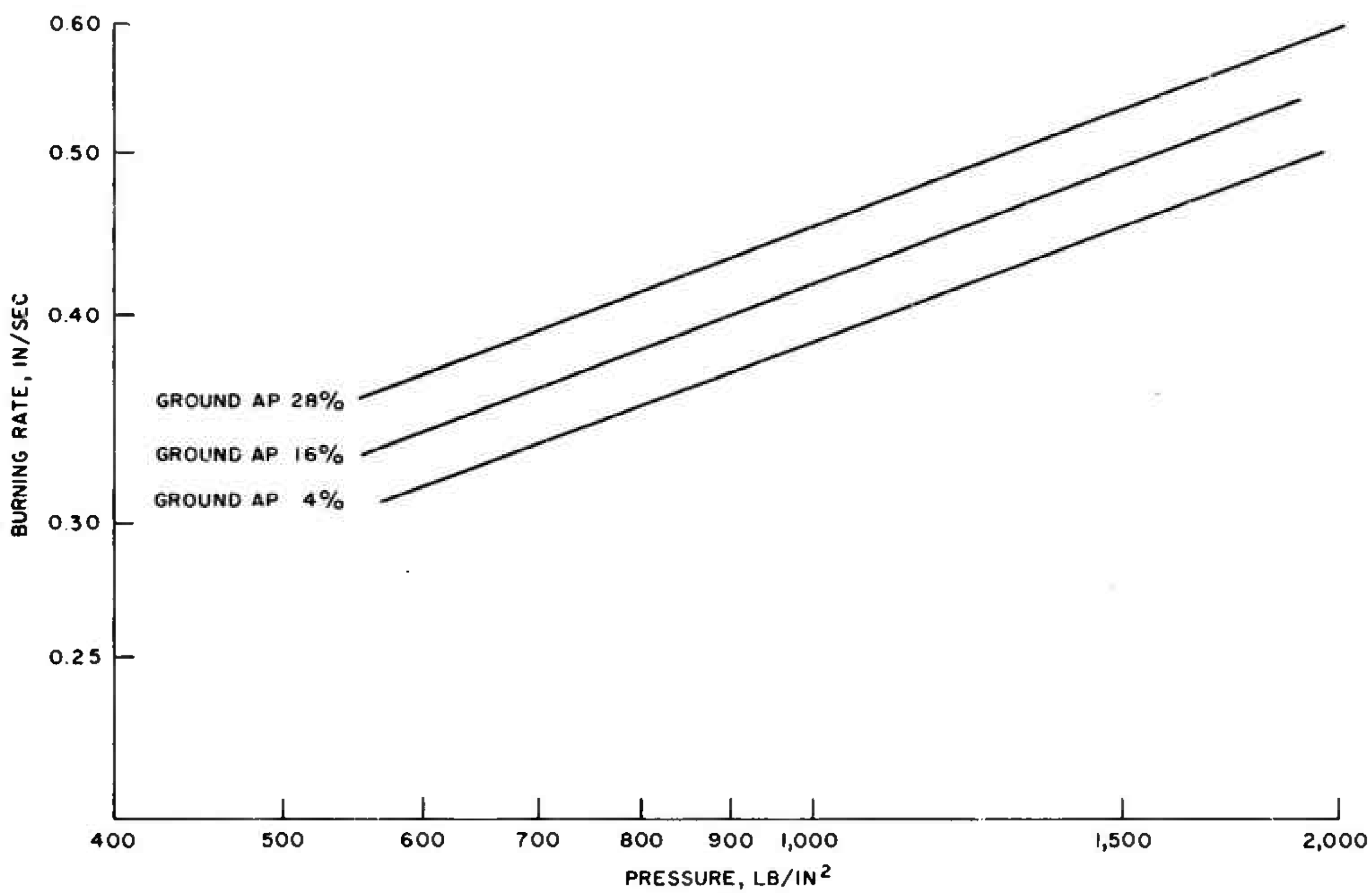

FIG. 11. Variation in Burning Rate With a Variation in Amount of Ground AP (Expressed as % of Total AP). **The 600μ Spherical AP is Constant at 25% and the Remainder is 180μ and Ground AP.**

HX-868 must be stored at temperatures below 0°F. At temperatures above room temperature, HX-868 degrades very rapidly ("degradation" means loss of active imine groups). At 225°F, HX-868 will be completely destroyed after 6 hours; at 180°F it will be 25 percent destroyed after 6 hours; and at 70°F it will be 15 percent destroyed after 64 days.

Adjusting the amount of crosslinker in the propellant (imine-to-carboxyl ratio) is the best method of quality control of the propellant physical properties. A change in raw material lots may result in a change of purity (hence a change in reactivity), and therefore an adjustment in imine-to-carboxyl ratio is usually necessary. In C-55A propellant, the imine-to-carboxyl ratio has varied from 0.60 to 0.85. Figure 12 shows the variation in tensile strength with a variation in imine-to-carboxyl ratio (I/C). All lots from 81 to 91 percent purity have given acceptable propellant.

Figure 13 is a plot of maximum tensile strength and elongation (at maximum tensile strength) versus cure time at 135°F for an imine-to-carboxyl ratio of 0.60 (the HX-868 used was 88 percent pure).

It is evident that most of the curing occures during the first 4 days and then the curve flattens out. The same curing pattern holds very well for imine-to-carboxyl ratios from 0.5 to 1. The values of tensile strength and elongation may differ greatly but the general shape of the curve remains the same.

This curve suggests that the optimum curing time (from a standpoint of aging or postcuring) is at least 10 days. Curing for this length of time will assure operation on the flat part of the cure curve. This means that the propellant has essentially reached full cure, and little change would be expected under normal storage conditions. However, if C-55A is used in a production motor, curing for 10 days is impractical due to the oven space and hardware required. For this reason, C-55A is usually cured 4 days with allowances made for the slight amount of postcuring that will occur.

PROPELLANT PROCESSING

C-55A propellant has been processed in vertical-type propellant mixers (Baker-Perkins, Bramley, and Day) ranging in size from 1 to 150-gallon capacity. No work has been done in horizontal mixers, since these are not available at NOTS. The processing steps are as follows:

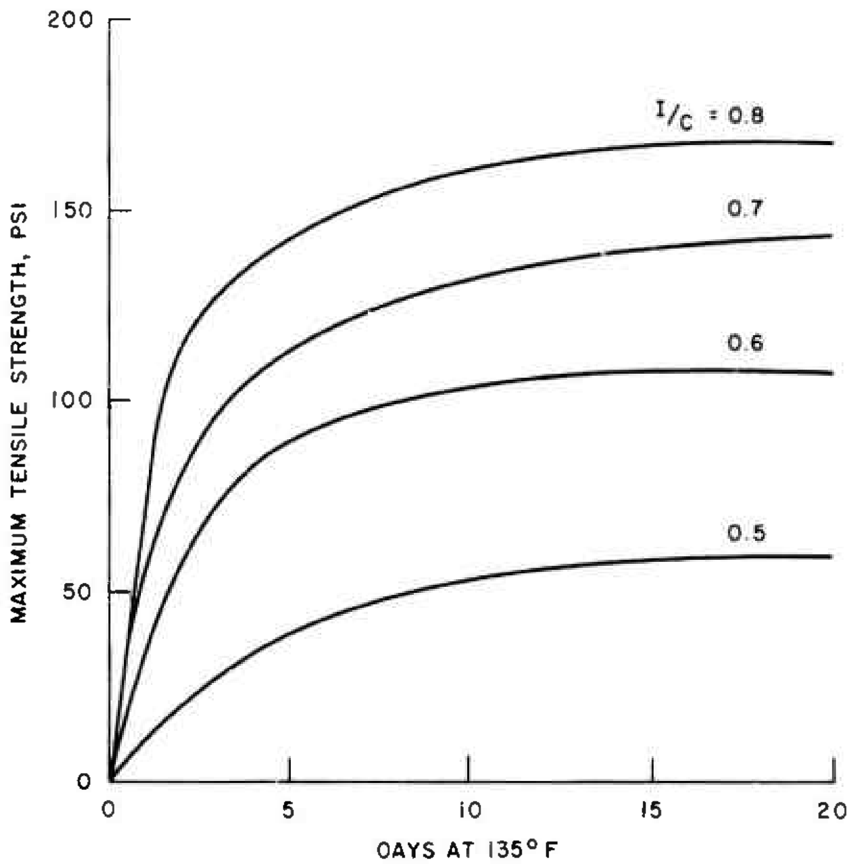

FIG. 12. **Maximum Tensile Strength Versus Cure Time for Different Values of I/C.**

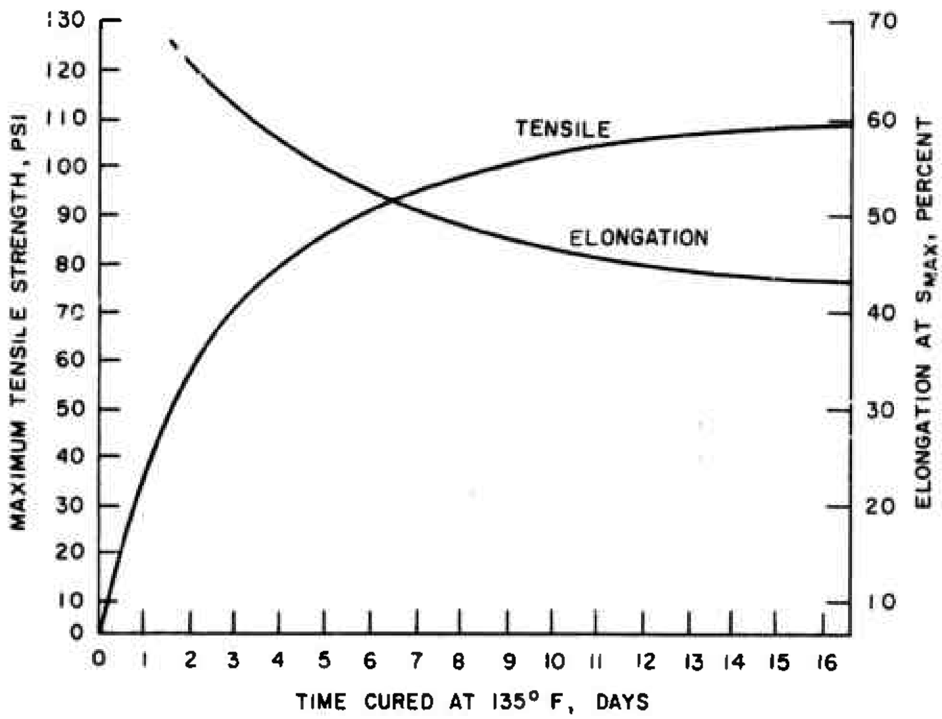

FIG. 13. **Maximum Tensile Strength and Elongation (at Maximum Tensile Strength) Versus Cure Time at 135°F.**

1. Place Butarez CTL-Type II, Al H-5, and yellow iron oxide into the heated mix pot (165°F jacket temperature). Retain some binder to add with the HX-868;

2. Mix the above ingredients for 10 minutes under 0.5 to 5 mm Hg absolute pressure;

3. Remotely add AP (preheated in 180°F oven) continuously while mixer is running at ambient pressure. This takes from 5 to 20 minutes depending upon the size of the mix. For small mixes the AP is added locally in steps of 1/2, 1/4, and 1/4 AP total weight with 3 minutes mixing time between each addition;

4. Scrape down mixer and then mix an additional 3 minutes;

5. Add HX-868 and the remainder of the binder. Mix 25 minutes. Final mix temperature should be 155 to 165°F. The final mixing cycle is under vacuum.

Scrape down (step 4) is primarily for safety reasons. It assures that there are no lumps of AP adhering to the mixer blades and sides which may come into contact with the concentrated crosslinker. The mixing of an equal weight of binder and HX-868 (step 5) is also for safety reasons. The binder, when mixed with the HX-868, provides protective coating in the event there is a hot, dry pocket of AP in the mix. Unmixed AP and concentrated HX-868 might start a fire.

The HX-868 may be added to the mix as either a liquid or a solid (depending on the purity of the HX-868). The HX-868 must not be heated above room temperature prior to adding to the mix. It may be added cold, directly from the storage freezer.

After the final mix cycle, the propellant viscosity is measured with a Brookfield viscometer.[10] The viscosity falls in the range of 400,000 to 600,000 centipoises at 160°F. The useful pot life of the propellant is 3 hours or more depending on the amount of cooling that takes place and the size of the motors that are being cast. Pot life is not limited as much by crosslinking in the pot as it is by cooling of the propellant while it remains in the pot.

Prior to casting, the liquid propellant is given an in-process heat of explosion test. The heat of explosion must be 1,545 ± 20 cal/g. C-55A propellant is always vacuum cast. Using vacuum techniques, extremely good bubble free grains of all sizes and shapes can be cast. The in-process heat of explosion test provides a reliable check on the composition of the propellant prior to casting. If error occurred in

[10] Brookfield Engineering Co., Inc, Stoughton, Mass.

the weighing or addition of propellant ingredients, it will be revealed by this test. Figure 14 is a quality control chart showing the heat of explosion measurements made on C-55A propellant. The points that fall outside of the acceptance limits will normally represent an error in propellant composition due to weighing or addition of ingredients. The data shown in Fig. 14 are the result of two laboratory measurements per batch; therefore, experimental error in taking the heat of explosion will at times account for a batch of propellant appearing to be out of specification. If an error in the measurement itself is suspected (indicated by a large range in the two measurements), then additional heat of explosion tests are made.

If C-55A propellant is used in a production motor, it is advisable to attempt to check burn rate prior to casting. This may be accomplished by making a liquid strand burning measurement. Liquid strand burning of C-55A has not been tried at NOTS, so it is not known if a good correlation exists between uncured and cured.

CASE BONDING

C-55A propellant is used in a case bonded grain configuration. The present method of preparing a motor case for casting consists of the following steps:

1. Degrease motor case with solvents
2. Grit-blast to roughen interior surface and reclean with solvent
3. Install boot or heat barrier, if applicable
4. Coat surface with Stanley primer
5. Paint or spin on LC-2 liner and cure
6. Cast propellant

LC-2 liner is simply Butarez CTL-Type II binder cross-linked with MAPO (imine/carboxyl = 2) manufactured by Inter-Chemical Corp.[11] and filled with 30 percent by weight medium thermal carbon black. LC-2 is normally applied by spinning the motor case while applying heat. However, for some applications, such as the head-end of a motor case, the liner is made thixotropic by the addition of Cab-O-Sil[12] so it will not flow, and is then painted onto the desired surface.

[11]Inter-Chemical Corp., Commercial Development Department, New York 36, N. Y.

[12]Godfrey L. Cabot Inc., Los Angeles 5, California

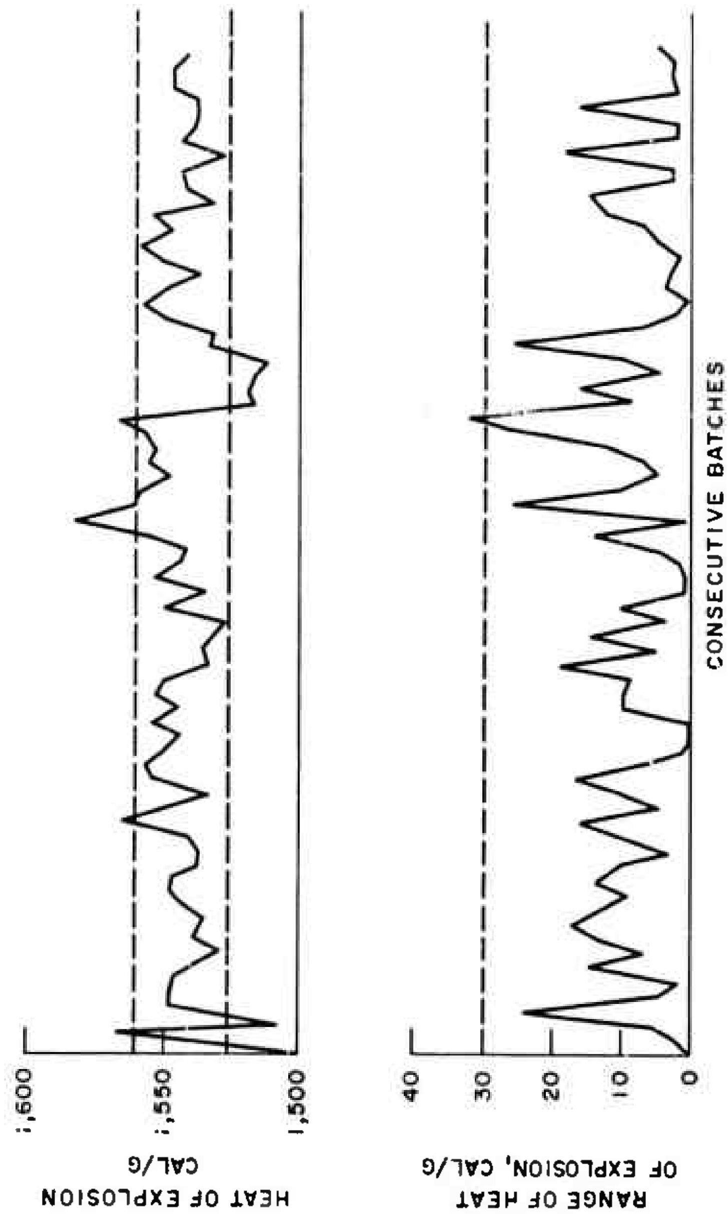

FIG. 14. Heat of Explosion Measurements, C-55A Propellant.

The LC-2 liner thickness is normally 0.040 ± 0.010 inch. A partial liner cure is obtained after 12 hours at 180°F. The propellant is then cast onto the liner and both receive a cure of 3 to 4 days at 135°F.

The bonding of C-55A propellant to the LC-2 liner is completely adequate. The tensile strength of the propellant-to-liner bond is in the same range as the tensile strength of the propellant (80 to 90 psi). The bond generally exhibits a cohesive failure in the propellant. This means that when the bond fails, a thin layer of propellant adheres to the liner. However, it is believed that this failure point represents a weakened area in the propellant. In other words, a thin layer adjacent to the liner exhibits less than normal physical properties and therefore failure occurs in this area.

CONCLUSIONS

C-55A is representative of present state-of-the-art composite propellants. It is readily processed in standard vertical mixers. C-55A is easily case bonded and gives excellent bubble-free grains when cast under vacuum.

C-55A exhibits good thermal stability. This propellant is comparable to other highly loaded rubber base propellants in terms of import and friction sensitivity and should be handled with care. Particular care should be given to avoid anything which may result in pinching or scraping the propellant between two hard surfaces. Machining the propellant is not unusually hazardous but must always be done remotely.

C-55A exhibits very good physical properties and is therefore suitable for use in a great variety of motors of different sizes and shapes. C-55A is sensitive to moisture and exhibits rapid degradation in physical properties at 180°F. However, it retains good physical properties at temperatures as high as 135°F, if not subjected to high humidity. Motors using C-55A propellant should have a moisture seal.

C-55A has ballistic properties which make it desirable from the standpoint of both performance and ease of motor design. The delivered I_{sp} of 245 seconds at standard conditions is 92.5 percent of theoretical. The burning rate is very reproducible, and the moderate burning rate exponent of 0.4 allows the use of conventional grain design. Reproducibility of C-55A ballistic properties can be controlled by the conventional method of adjusting the AP particle size distribution.

Appendix A

LIST OF SPECIFICATIONS

Butarez CTL-Type II	MIL-P-23942(WEP) Polybutadiene, Linear, Carboxyl Terminated, Types I and II (19 August 1964)
HX-868	See Manufacturers Specification, Minnesota Mining and Manufacturing Corp., 367 Grove Street, St. Paul 1, Minnesota
Yellow Iron Oxide	MIL-F-23938(WEP) Ferric Oxide for Propellant Grains Mark 75 and Mark 76 (19 August 1964)
Aluminum Powder(Al-H5 Type 1)	MIL-A-23950(WEP) Aluminum Powder, Spherical (19 August 1964)
Ammonium Perchlorate (NH_4ClO_4)	MIL-A-192A Ammonium Perchlorate, Technical (20 October 1960) MIL-A-23442A(WEP) Ammonium Perchlorate (3 March 1965)
MAPO	4535/FBF:baj Reg. 4535-25-66 Quality Control Acceptance Procedure for MAPO TRIS 1-(2-Methyl) Aziridinyl Phosphine Oxide
CAB-O-SIL	See Manufacturers Specification, Godfrey L. Cabot, Inc., 718 Texaco Building, 3350 Wilshire Blvd., Los Angeles 5, California
Carbon Black	MIL-C-307B Carbon Black, Dry (For Use In Explosives) 30 April 1965

ABSTRACT CARD

U. S. Naval Ordnance Test Station
<u>Characterization of C-55A Propellant</u> (U), by M. Frank Pickett. China Lake, California, NOTS, April 1966. 38 pp. (NAVWEPS Report 9013, NOTS TP 3997), CONFIDENTIAL.

ABSTRACT. The C-55A propellant is a cast composite utilizing a carboxyl-terminated-polybutadiene binder cross-linked with a trifunctional imine. C-55A propellant delivers a specific impulse (I_{sp}) representative of state-of-the-art aluminum-ammonium perchlorate (Al-AP) composite propellants. It has excellent physical

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properties and is ideal for use in case bonded motors. C-55A propellant will withstand storage temperatures up to 135°F with little change in physical properties if the propellant is protected by a moisture seal. C-55A propellant is currently being used in a rocket motor developed by the U. S. Naval Ordnance Test Station (NOTS), China Lake, California designated NOTS Mod 401A rocket motor.

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14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
COMPOSITE PROPELLANT LC-2, LINER BINDER, CARBOXYL-TERMINATED-POLYBUTADIENE ALUMINIZED PROPELLANT						

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